IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF:	CONFIRMATION NO. 9545
DONALD L. RYMER ET. AL.	CASE NO.: AD6856 US PCT
SERIAL NO.: 10/501,598	GROUP ART UNIT: 1796
FILED: JULY 13, 2004	EXAMINER: WILLIAM K. CHEUNG

FOR: LOW-COLOR PVB SHEET AND A PROCESS FOR MAKING SAME

DECLARATION UNDER 37 CFR1.132

Commissioner for Patents
P.O. Box 1450
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Sir:

I, Donald L. Rymer, declare and state:

I am a named co-inventor on the subject patent application and am an employee of E. I. du Pont de Nemours and Company (DuPont), the assignee of the subject patent application.

In 1966 I was awarded a Bachelor of Science degree in Chemistry from Salem College.

In 1968, I was awarded a Master of Science degree in Organic Chemistry from Ohio University in Athens, Ohio.

I have worked for DuPont for over 39 years in a variety of manufacturing technology and research assignments.

I am currently a Technology Fellow conducting product and process research for the Packaging and Industrial Polymers (P&IP) business and work in the field of poly (vinyl butyral) (PVB).

I have worked in the PVB field for over 30 years developing polymer syntheses, troubleshooting manufacturing problems, and improving our product as it is incorporated as an interlayer in glass laminates.

I have been a lead technology person in starting up PVB labs and processes outside the U.S. and continue to be involved in the design of new products.

I am also a named co-inventor on the following patent applications:

Patent/Publication Number	Grant/Publication Date	Official Title (English)
US-2006-0183833-A1	08/17/2006	LOW-COLOR STIFF PVB LAMINATES
US-2005-0288429-A1	12/29/2005	A PROCESS FOR CONTROLLING POLYVINYLBUTYRAL PHYSICAL PROPERTIES BY CONTROLLING STEREOCHEMISTRY OF SAME
US-2005-0234185-A1	10/20/2005	POLYVINYLBUTYRAL INTERLAYER SHEET WITH IMPROVED ADHESION TO GLASS AND A PROCESS FOR PREPARING SAME
US-2006-0008648-A1	01/12/2006	POLYVINYLBUTYRAL INTERLAYERS HAVING SUPERIOR ACOUSTICAL PROPERTIES AND METHOD OF PREPARING SAME
US-2008-0157426-A1	07/03/2008	PROCESS AND APPARATUS FOR REDUCING DIE DRIPS AND FOR CONTROLLING SURFACE ROUGHNESS DURING POLYMER EXTRUSION

The claims of the subject application are directed to a process for preparing a low color, polyvinyl butyral sheet for use in the manufacture of glass laminates. Claim 1 is typical of the independent claims and recites as the first two steps:

(I) admixing polyvinyl alcohol, butyraldehyde, an acid or mixture of acids, water, and sodium dialkyl sulfosuccinate;

(II) stabilizing the mixture obtained in step (I) by (a) raising the pH of the mixture to at least pH 10, (b) isolating the polyvinyl butyral resin composition by draining the liquid, and (c) washing the polyvinyl butyral resin composition with neutral pH water;

I am aware that the claims of the subject patent application stand rejected under 35 USC 103(a) as obvious over a number of patent documents including Degeilh (US 4,696,971). Degeilh teaches that the described process should be carried out with sodium dialkyl sulfosuccinate and neutralizing to pH of no more than 5.

All of the work described herein was carried out by me or under my supervision.

The data in this Declaration is based upon actual manufacture of PVB and PVB laminates, and focuses on the neutralization step when dioctyl sulfosuccinate (DOSS) is used. It includes my analysis of PVB laminates made from PVB produced by DuPont using sodium dioctyl sulfosuccinate (DOSS) wherein the neutralization is carried out at different pHs.

One laminate sample representing the invention was made using a flake neutralized at a ph of 10.2. A comparative PVB laminate was made using a flake neutralized at a ph of 8.5. Both were made using DOSS and were made on the same equipment using the same parameters (except neutralization was carried out to the pHs described) by the following steps.

As explained below, laminates were not made using flake neutralized at lower pH because of concern over flammability in the dryer. In addition, the PVB made at pH 4.5 was observed to have higher color than PVB neutralized at higher pHs.

The first step of the process used to make the PVB was admixing polyvinyl alcohol, butyraldehyde, an acid or mixture of acids, water, and sodium dioctyl sulfosuccinate at 90°C. The next step involved stabilizing the mixture obtained in the first step by raising the pH of the mixture to 10.2 (invention examples) or 8.5 (comparative example). This was done in two stages. First the material was taken to a pH of 4.5 to avoid a chemical reaction reversal. During this step the excess butyraldehyde was sparged out to reduce the formation of 2-ethy-2-hexenal. Next the reaction batch of the invention was taken to a pH of 10.2 to avoid excess formation of the fine PVB "gel" which we see in the laminates. The pH was taken to 8.5 for the comparative example. The invention and comparative sample materials were then further processed by isolating the polyvinyl butyral resin composition by draining the liquid, and washing the polyvinyl butyral resin composition with neutral pH water. That was followed by drying the PVB resin and extruding the PVB resin (flake) with plasticizer and additives at a temperature of from about 209°C to about 210°C to obtain PVB sheeting. The flake neutralized at the two different pH levels was extruded into sheeting using the process described in US 3,153,009 Example (III). From this sheeting standard glass laminates were prepared and examined for clarity.

Here it is noted that while the pH was first taken to a pH of 4.5 it was not feasible to make laminates from that material. That material was determined to be thermally unstable and pose a processing risk of fire in the dryer. Hence, for safety reasons a comparative laminate sample was not prepared from the material that was at 4.5 pH.

I note that in my observations of the three materials, the material that was neutralized at 4.5 pH was the most highly colored and, thus, in addition to its thermal instability, it is my opinion based upon my experience in the art that this material was less suitable for making laminates of the two materials. In addition, it is my opinion (as is confirmed with the data) that the materials neutralized at pH 10.2 had better color than and was better suited for making laminates than the material neutralized at pH 8.5.

After the laminates were prepared I observed both of them. The invention sample laminate was very clear. The comparative sample laminate showed defects. These differences were magnified when viewed under high intensity light, which is common windshield test used in the industry to observe clarity.

I also evaluated the samples under a microscope using UV light and counting and sizing device. Attached is a graph showing the gel counts. The lighter line (blue) to the left, when present, represents the invention and the darker line (red) (to the right) represents the comparative sample. The comparative sample had larger amount and larger size gel particles than the sample representing the invention. Higher quantities of and larger sized gel particles are unacceptable because they are easier to see and scatter light. Thus, the invention provided significantly superior results over the comparative sample.

It is noted that since the material prepared at pH 4.5 had more color than other two samples when observed by eye, it is my opinion that had there been no safety concern and had a laminate been prepared, that sample would have had more gel particles than both the samples prepared at pH 8.5 and pH 10.2.

Given the above, it is my opinion that the invention, which involves use of sodium dialkyl sulfosuccinate (e.g., DOSS) and neutralizing to pH or at least 10, provides better clarity laminates than laminates prepared from PVB sheet made using a process involving sodium dialkyl sulfosuccinate and a neutralization step at lower pHs. The comparative sample neutralized at a ph of 8.5 had more gel counts and was not as clear, and based upon my knowledge in the pertinent art and the results obtained with that sample, as well as based upon my observations of the material prepared at pH 4.5 was not safe to process through the dryer, my expectation is that even worse results would have been obtained had the pH been lower. It is also my opinion based upon my knowledge in this art and based upon the work carried out, that the better clarity obtained with the claimed invention would not be expected based upon the Degeilh and the other documents cited in the rejections.

Based upon my experience in the pertinent art and the work carried out, it is my opinion that the results support that unexpected results are obtained with the invention as claimed and, thus, are commensurate in scope with the claims. Thus, it is my opinion that the invention provides unexpected results over the process described in Degeilh and that these results are significantly better than would be obtained with the process of Degeilh.

All statements made herein of my own knowledge are true, all statements made herein based on information and belief are believed to be true, and further that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,
/Donald L. Rymer/

Dated: April 20, 2009

Attachment - Gel Counts of PVB Laminates